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EFFECT OF Zr OR Cr CONTENT ON THE FRACTURE TOUGHNESS OF THE HIGH PURITY Al-Zn-Mg ALLOYS

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16. Abstract

Relationship between the microstructure and the fracture toughness of high purity Al-Zn-Mg alloys, to which the Zr and Cr were added changing the quantity, was investigated. Adding the Zr content, it is observed that the grain size becomes smaller and the fracture toughness becomes bigger as the increased quantity. But as for the fracture toughness, it's maximum is seen when the Zr content is added with 0.18%. This is why, in case of Zr content is little in quantity, recrystallization is not controlled and the grain size becomes large, consequently the concentration in grain boundary localization occurs and it gives a intergranular fracture. In case of the addition more than 0.18% Zr, the distributive density of η' phase becomes low as soon as the large dispersoids appears in the matrix, and the fracture toughness decreases. As for the Cr content, the tensile strength falls in accordance with the increase of the added quantity, but the fracture toughness increases up to 0.32%. Because of the prior precipitating of η phase in the Cr despersoid uniformly in matrix. In comparison with Zr and Cr content, Zr content is superior to Cr content on the tensile strength. On the other hand, Cr content is superior to Zr content on the absorbed energy.

Keywords: Al-Zn-Mg alloy, fracture toughness, Zr or Cr dispersoid, precipitation particle

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Eeji Kato*, Yoshisada Ueda* and Hideshi Mori**

Relationship between the microstructure and the fracture toughness of high purity Al-Zn-Mg alloys, to which the Zr and Cr were added changing the quantity, was investigated. Adding the Zr content, it is observed that the grain size becomes smaller and the fracture toughness becomes bigger as the increased quantity. But as for the fracture toughness, it's maximum is seen when the Zr content is added with 0.18%. This is why, in case of Zr content is little in quantity, recrystallization is not controlled and the grain size becomes large, consequently the concentration in grain boundary localization occurs and it gives a intergranular fracture. In case of the addition more than 0.18% Zr, the distributive density of η' phase becomes low as soon as the large dispersoids appears in the matrix, and the fracture toughness decreases. As for the Cr content, the tensile strength falls in accordance with the increase of the added quantity, but the fracture toughness increases up to 0.32%. Because of the prior precipitating of η phase in the Cr despersoid uniformly in matrix. In comparison with Zr and Cr content, Zr content is superior to Cr content on the tensile strength. On the other hand, Cr content is superior to Zr content on the absorbed energy.

Keywords: Al-Zn-Mg alloy, fracture toughness, Zr or Cr dispersoid, precipitation particle

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1. Introduction

High purity Al alloys have a defect that they are weak in the fracture toughness, though relatively strong in the tensile strength. This low fracture toughness is mainly caused by the existence of particles of impure elements [1]-[3]. In order to improve this defect, it has been tried to decrease impurity particles by using a high purity parent metal. However, because of the thermal treatment after processing, crystal grains increase in size and breakage between the grain boundaries occur, and consequently the fracture toughness decreases [4-5]. Therefore, we add Zr, Cr., etc. to suppress the increase in size of the crystal grains by thermal treatment after processing. However, addition of Zr and Cr forms insoluble chemical compounds [4], [6]

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in the process of homogenizing thermal treatment and thermal treatment after the processing. Dispersed particles of these chemicals of 0.05-0.5 μm in size exist in the grain boundaries and in the body.

Usually, fracture of Al-alloys is caused by ductile destruction and the behavior of this fracture goes through the stages of void formation, starting at the second phase grain in the alloy and the growth and assembling of the voids [7,8]. We assumed that the dispersed particles in the Zr-Cr group also play an important role in the formation and growth of the voids, and affect the fracture toughness, and investigated the fracture toughness and the microstructure of high purity Al-Zn-Mg alloys of various amounts of added Zr and Cr in the present work.

2. Experimental method

We added various amounts of Zr and Cr to the parent metals which were supposed to have Al 5% Zn 2% Mg. In this experiment, we used high purity source metals (99.99 Al, 99.9 Zn, 99.9 Mg, Al 5%, Zr: 0.10% Fe, Al-3.4% Cr: 0.13% Fe) in order to prevent mixing of impurities. We melted them and cast in a metal mold (30x60x110 mm) placed on a water cooled copper chill plate to obtain lumps of alloys. The chemical composition of these lumps is shown in Table 1. These molded alloys were thermally treated to make them homogeneous at 720 K for 12 hours and then water cooled. Subsequently, they were pressed between a heat gap by 30% at 573 K, and then pressed between a cold gap by 20% to obtain test specimens.

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In each test we used specimens of the shape shown in Figure 1 which were cut out in the direction of rolling. We carried out simple tests of the tensile strength, the standard V-notch Charpy test, and the fracture toughness by the gauge equipped Charpy test [9].

Table 1 Chemical composition of materials (wt%)

Material	Zn	Mg	Cu	Zr	Cr	Fe	Si	Al
A	4.49	1.89	0.25	0.07	—	0.04	0.01	bal
B	4.78	2.06	0.26	0.13	—	0.03	0.01	bal
C	4.66	1.86	0.29	0.18	—	0.04	0.01	bal
D	4.70	1.95	0.27	0.22	—	0.04	0.01	bal
E	4.74	1.95	0.26	—	0.17	0.04	0.01	bal
F	4.85	1.98	0.26	—	0.32	0.04	0.00	bal
G	4.80	1.97	0.26	—	0.61	0.04	0.01	bal

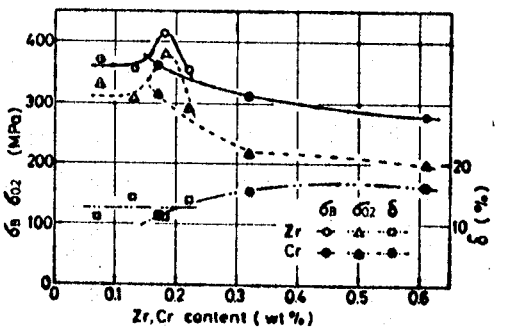


Fig. 2 Effect of Zr and Cr content on the tensile properties.

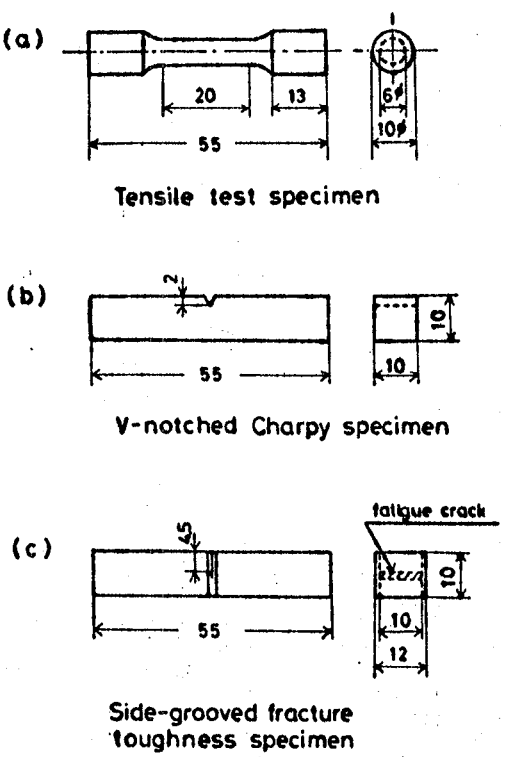


Fig. 1 Specimen geometries (mm).

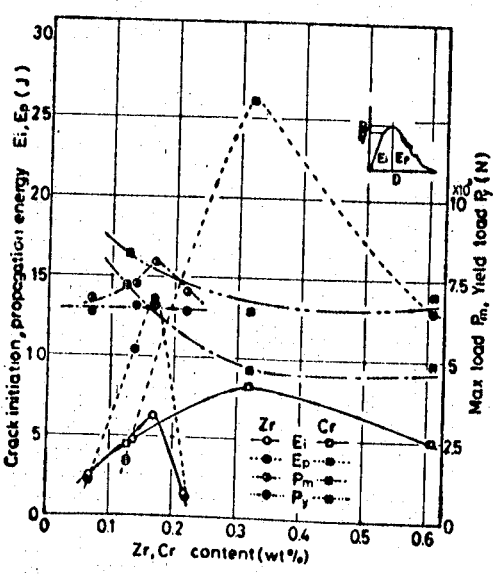


Fig. 3 Effect of Zr and Cr content on the Charpy V-notch test.

For the tensile strength test, we used the Instron type tensile strength measurement device (the strain speed 4.6×10^{-4}/s), and for other tests we used a 98J Charpy test device (the hammer speed, V notch test 4.4 m/s, the fracture toughness test 2.1 m/s). All the tests were done at room temperature.

The conditions for thermal treatment of these test specimens were that after solid solutions by 723K x 1 hr, full aging was done by 393 x 32 hrs.

We used optical and transmission type electron TEM microscopes for structure observation, a scanning electron microscope (SEM) for fracture surface observation and an X-ray photon microanalyzer (EPMA) and an energy dispersion type microanalyzer (EDXS) for particle analysis.

3. Experimental results and discussion

3.1 Relation between the tensile strength characteristics and the added amounts of Zr and Cr

Figure 2 shows experimental results of the tensile strength test when the amounts of Zr and Cr added were varied.

When Zr was added, both the maximum tensile strength (σ_B) and the 0.2% endurance ($\sigma_{0.2}$) gradually increased as the added amount increased. After reaching their maximum values at 0.18% addition, they suddenly decreased at 0.22% addition. The stretch (δ) did not vary significantly with the added amount.

When Cr was added, both σ_B and $\sigma_{0.2}$ decreased as the added amount increased. The value of δ increased up to the point where the addition was 0.32%, and then remained constant beyond the point.

Concerning the difference between Zr and Cr additions, the values of σ_B and $\sigma_{0.2}$ were higher when Zr was added and δ was slightly larger when Cr was added. /793

3.2 Relation between the fracture toughness and the added amounts of Zr and Cr

Figure 3 shows results of the V-notch Charpy test for obtaining the load-displacement curves when different amounts of Zr and Cr are added. In this case, we assumed that the absorption energy up to the maximum load (P_m) was the so-called crack initiation energy (E_i) and that after the maximum load the so-called crack propagation energy (E_p), and defined the yielding load (P_y) by the load at which the load-displacement curve significantly shifted from a straight line. Also, the displacement (D) was measured by using a potentiometer attached to the revolving axis of the Charpy test device.

Both E_i and E_p increased by the addition of Zr up to 0.18% addition. Beyond the point, both values rapidly decreased, especially E_p which decreased significantly. When 0.07% and 0.22% were added, the curves show high speed unstable destructions in which displacements beyond the maximum load are small. The P_m value increased up to the 0.18% addition, but the P_y value did not change noticeably.

When Cr was added, both E_i and E_p increased up to 0.32% addition.. The E_p value is especially high at 0.32%.

In comparing the Zr and Cr additions, we observe a large difference in the absorption energy and the value is especially high when Cr is added. In particular, the E_p value is almost twice as large when Cr is added, compared to the case of the Zr addition.

3.3 Relation between the fracture toughness and the added amounts of Zr and Cr

Figure 4 shows results of simplified evaluations of the fracture toughness measured by the Charpy test device when different amounts of Zr and Cr were added.

When Zr is added, the fracture toughness (J_{IC}) tends to increase up to the 0.18% addition point, and then rapidly decreases beyond the point.

When Cr is added, the J_{IC} value rapidly increases with the amount of Cr, reaches its maximum at 0.32% addition and then gradually decreases.

The difference between Zr and Cr additions is that the maximum value is larger when Cr is added. This agrees with the results of the absorption energy measured by the V-notch Charpy test.

3.4 Microstructure and observation of the fracture surface

As described above, when Zr and Cr are added to the present alloy, the fracture toughness can be increased. However, adding excess amounts results in decrease of the fracture toughness. This is probably caused by changes in the microstructure by Zr and Cr additions. Figure 5 shows the photographs of the structures of each specimen observed by optical microscope.

When Zr is added, the crystal grain size decreases as the added amount increases. The size in specimen C (0.18% Zr; the average grain diameter 31.8 μm) is about one-half that of specimen A (0.07% Zr, the average grain size 64.6 μm). However, the size in Specimen D (0.22% Zr, the average grain size 29.3 μm) is almost the same as in specimen C.

When Cr is added, the grain size in specimen F (0.32% Cr, the average grain size 17.6 μm) is about half that of specimen E (0.17% Cr, the average grain size 41.9 μm). Specimen G (0.61% Cr) which has a larger percentage of Cr, does not show clear-cut grain boundaries and, therefore, the grain size could not be measured. This is probably because we carried out a homogenizing thermal treatment in brine bath, and therefore the speed of the temperature increase was too fast for Cr group chemical compounds to uniformly precipitate, and consequently, precipitation occurred mostly near the grain boundaries [10].

Figure 6 shows photographs of the fracture surface observation when Zr was added. In specimen A, most of the fracture occurred at the grain boundaries. Also, since the crystal grain is large, the fracture surface unit is large. In specimen B, dimple and flat parts are mixed on the fracture surface. At edges of the flat part, dimples which appear to be particles existing at the grain boundary are observed [9]. In specimen C, most of the surface is occupied by dimples. However, fracture surfaces which appear to have the edge at the grain boundary, are partly observable (b). The average depth and the diameter of the dimple part in specimen B are 22.9 μm (average diameter) and 7.9 μm (average depth) and therefore, the unevenness of the fracture surface is relatively small. In specimen C, the average diameter 22.1 μm and the average depth 13.9 μm, which compared with specimen B, is more uneven, although the diameter does not change appreciably. In specimen D, the fracture surface is mostly flat. However, compared to specimen A, the fracture surface unit is smaller. This is probably because two are different in the crystal grain size, and the size in specimen D is about half that of specimen A.

The fracture surface when Cr was added (Figure 7) is mostly flat in specimen E. However, in specimens F and G, the fracture surfaces are of the rolled type with deep dimples. The average diameter and the average depth of the dimple is 17.1 μm, and

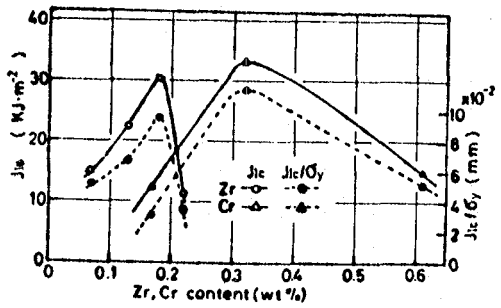


Fig. 4 Effect of Zr and Cr content on the fracture toughness.

and 6.5 μm, respectively in specimen F, and 14.9 μm and 5.8 μm, respectively in specimen G.

As seen from the above discussion, the fracture toughness tends to increase as the crystal grain size decreases. However, even if the crystal grain size is the same, the fracture toughness is not the same. This is probably because of different precipitations in the grain and at the grain boundaries. Therefore, we studied the peak aged precipitation phase and dispersed particles in the grain and the grain boundary. Figure 8 is a photograph of TEM observation when Zr is added. In B and C specimens, we do not observe differences in the peak aged phase, conditions of Al_3Zr and distribution concentration. The size of the peak aged precipitation phase in the grain in this case is less than 0.01 μm. However, in specimen D, a considerable amount of precipitation of large grains (0.2-0.3 μ) which appear to be precipitates of the η-phase and dispersed particles of the Zn group below is observed and also the distribution density of the peak aged, fine particle precipitates is smaller than in specimens B and C. On the other hand, the precipitation at the grain boundaries is that the number of precipitates at the grain boundaries increases as the Zr amount increases, and the PFZ width narrows (the width is ~1.2 μm in specimen B, ~0.5 μm in specimen C and ~0.2 μm in specimen D).

The fracture toughness in destruction by rolling is the value of resistance which an object shows when edges of defects in the material dull, and voids, originating at the secondary phase particle near the fractured edges, are formed , grown larger, assembled and then develop into fractures when an edge of the fracture edge is relaxed, and the maximum tensile stress is generated at the forward edge of the fracture. Consequently, formation and growth of voids are accelerated at this location [1,2]. Therefore, we carried out identification particles which

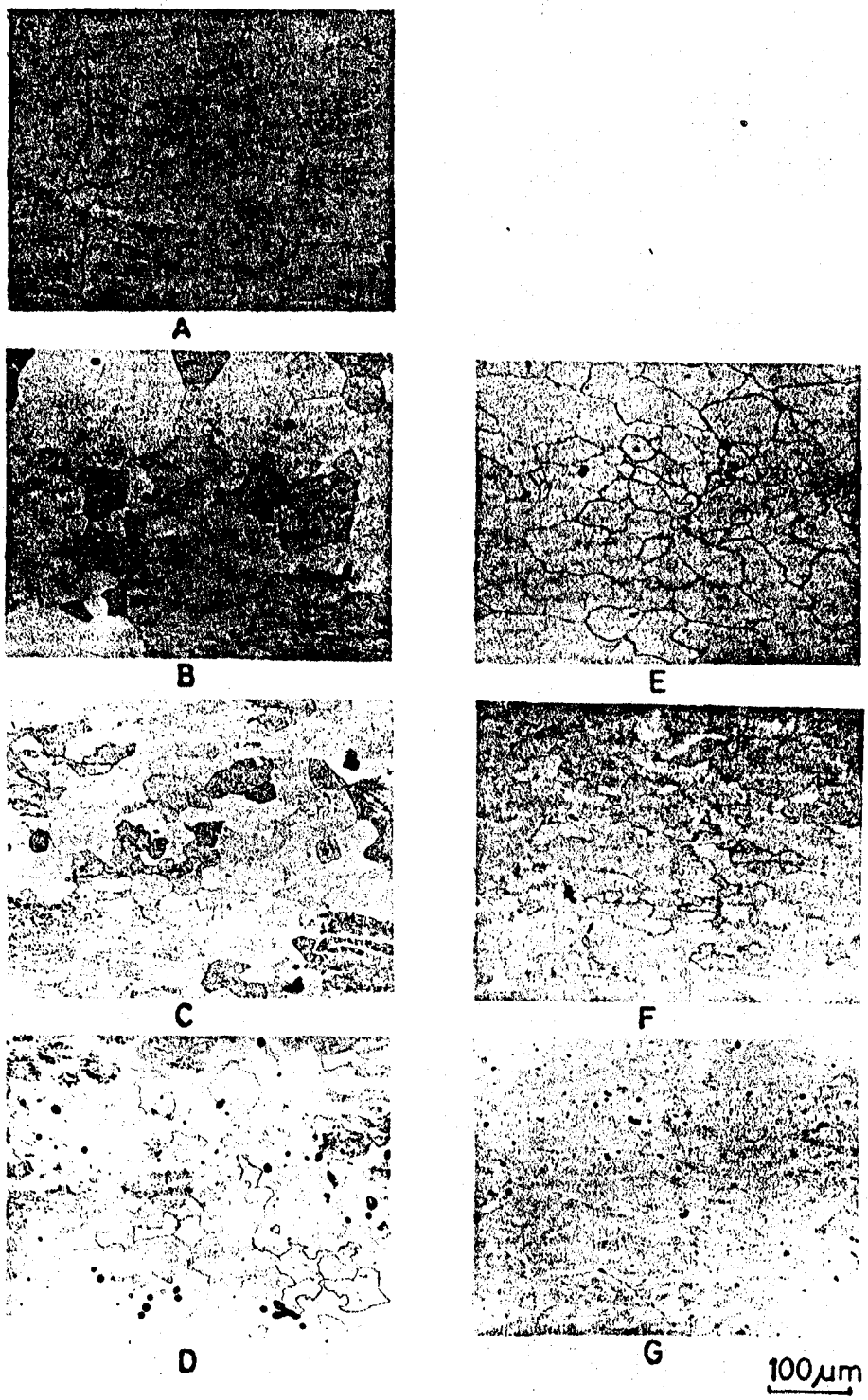


Fig. 5 Effect of Zr and Cr content on the microstructure of Al-Zn-Mg alloys.

accelerate void formations in this region. When Zr was added, there are two kinds of particles which were at the bottom of the dimple at the forward edge of a fracture, as seen in Figure 9; namely, particles at the bottom of a large dimple and those at the bottom of a small dimple. (We could not distinguish particles inside from those at the grain boundary when grain identification shown in Figure 9 was done. Particles at the grain boundary are those included in the specimens for microscopic observation). Particles at the bottom of a small dimple appear to be dispersed particles of the Zr group (Al_3Zr of radius less than 0.01-0.02 μm, and the peak aged precipitates (the η-phase, the diameter less than 0.01 μm). However, we would not identify them. Particles at the bottom of a large dimple appear to be inclusions (Al-Fe group compounds and the diameter 0.2-0.5 μm) since the grain diameter is about 0.2-0.5 μm. The particle size which controls the fracture toughness is, according to Kawabata, et al. [3,4] larger than ~0.4 μm. Therefore, we infer that the existence of such large particles in a large amount at the grain boundary or inside the grain determines the fracture toughness. Viewing the TEM photographs, there are only particles smaller than 0.01 μm in diameter inside the grain, and it is inconceivable that they cause void formation, and thus they only affect fracture propagation. On the other hand, at the grain boundary, there are particles of 0.2-0.3 μm and void formation results from splitting of the particle or detachment of the particles from the boundary. Also, since there exists PFZ of small deformation resistance along the grain boundary, void formation and growth become easier, and this seems to control destruction. However, if the Zr content is high, as in specimen D, there are large particles inside the grain as well as at the grain boundary and they seem to lower the fracture toughness. On the other hand, if the Zr content is low, as in specimen A, movement of the grain boundary is not suppressed and the crystal grains are large, and this causes the grain boundary fracture and lowering of the fracture toughness. Difference in the fracture toughness in specimens B and C seems to be caused by the size and the amount of the

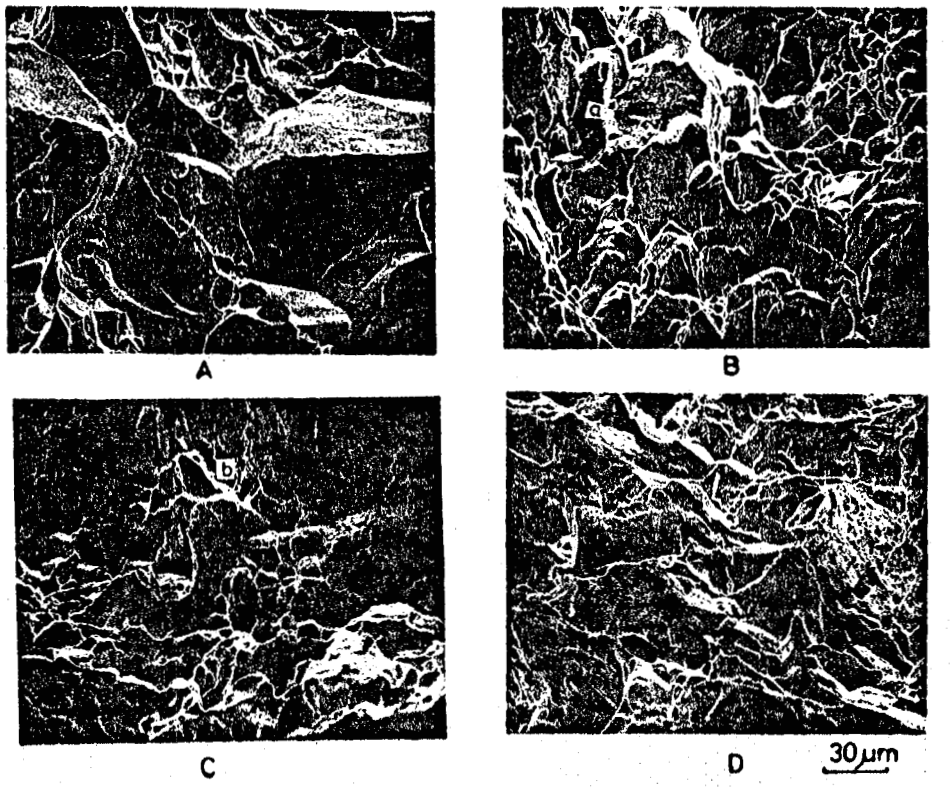


Fig. 6 SME fractographs of the peak-aged Zr content materials.

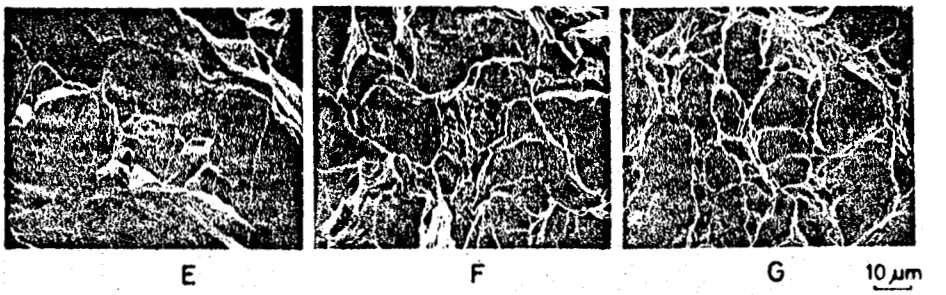


Fig. 7 SEM fractographs of the peak-aged Cr content materials.

precipitation phase at the grain boundaries, and the mutual interaction of different PFZ widths. However, we hope to investigate the quantitative determination of the structure regarding this point in the future.

When Cr is added, not only the peak aged, fine precipitation particles but also particles of 0.2-0.4 μm, which appear to be precipitated on the Cr group dispersed grains, also exist inside the grain. The latter are the same or larger in size, compared

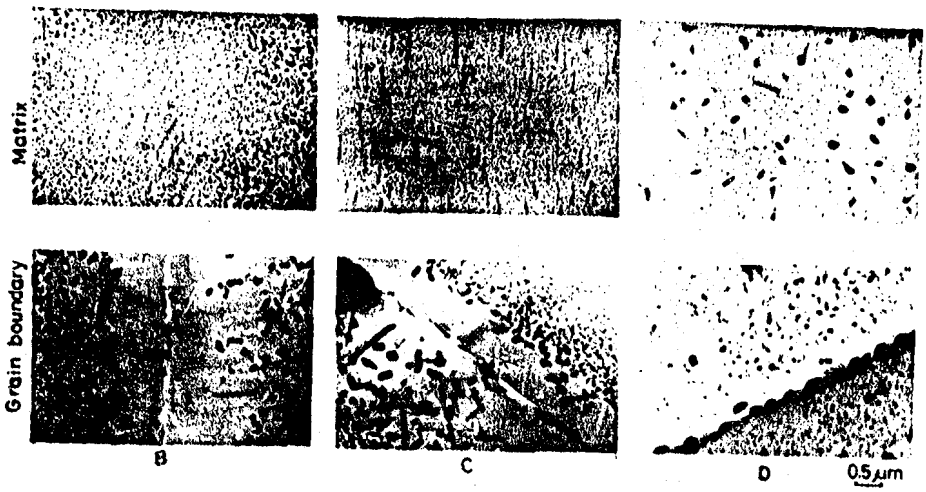
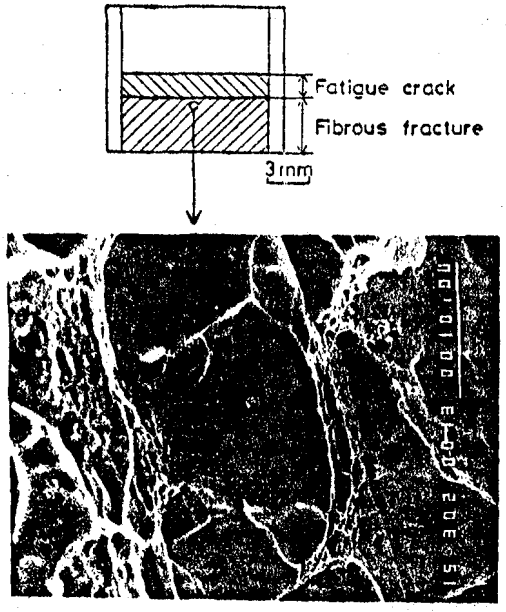


Fig. 8 TEM microstructures of the peak-aged Zr content materials.



	Al	Zn	Mg	Si	Fe	Zr
Grain boundary particle	77.19	7.48	3.70	0.93	9.84	0.74
Particle in dimple bottom	71.72	8.37	4.86	2.81	11.90	0.34
	68.99	7.23	3.43	0.44	16.01	0.42

Fig. 9 EPMA analysis of inclusions at the region ahead of crack tip (sample C material).

to particles precipitated at the grain boundary (Figure 10). These particles can cause void formation. Particles at the bottom of large dimples at the forward edge of a fracture are mainly the peak aged precipitates containing CR (Figure 11) and different from the case of Zr addition. Consequently, it is probably true that fracture by addition of Cr is related to particles inside the grain.

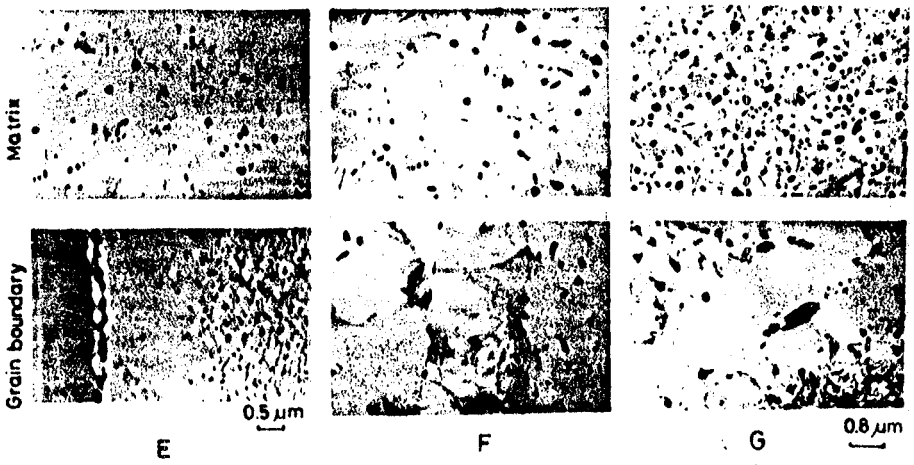
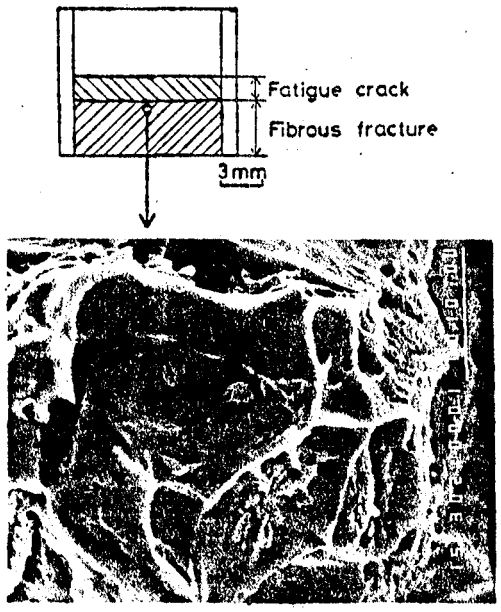


Fig. 10 TEM microstructure of the peak-aged Cr content materials.



	Al	Zn	Mg	Si	Fe	Cr
Grain boundary particle	66.03	8.64	7.34	2.54	14.19	0.11
Particle in dimple bottom	58.12	10.54	11.16	14.92	0.27	3.27

Fig. 11 EPMA analysis of inclusion at the region ahead of crack tip (sample F material).

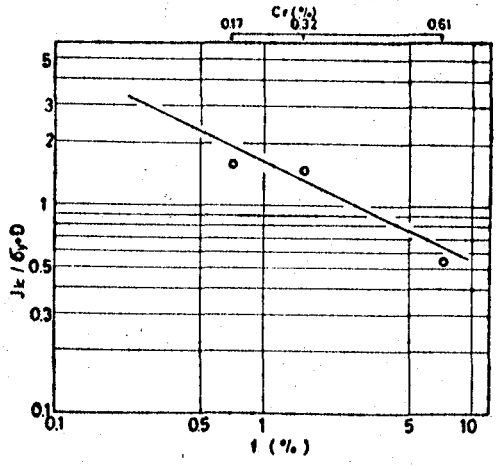


Fig. 12 Relationship between $J_c/\sigma_y \cdot D$ and volume fraction f.

Generally, the fracture strength of the secondary particle control type type is given by the following equations, using the diameter and the volume ratio of the secondary particle [15,16].

$$J_{IC}=G_{IC}=[2\sigma_y \cdot (1-r^2)(\pi/6)^{1/3} \cdot D]f^{1/3} \qquad (1)$$

where σ_y is the yielding stress, f the volume ratio of the secondary particle, D the diameter of the particles participating in fracturing and r the Poisson ratio. We show the relation between $J_{IC}/\sigma_y \cdot D$ and f in Figure 12. The $J_{IC}/\sigma_y \cdot D$ value decreases and f increases and the slope is $\sim -\frac{1}{3}$, and correlates well with equation (1). Therefore, we consider that the fracture strength of Cr added materials is controlled by the peak aged particles precipitated on the Cr-group dispersed particles inside the grain.

4. Conclusions

We investigated the fracture strength and the microstructure by adding different amounts of Zr and Cr to high purity Al-Zn-Mg-alloy and obtained the following conclusions:

(1) By adding Zr, the fracture strength is the maximum when the addition is 0.18%. The tensile strength also reaches the highest at the same point.

This is because by adding less than 0.18%, movement of the grain boundary is not suppressed and the diameter of the crystal grain becomes large. This causes a locally concentrated stress and promotes the grain boundary fracture. If the addition is more than 0.18%, large dispersed particles appear inside the grain and simultaneously the distribution density of the η' phase is lowered, resulting in lower fracture toughness values.

(2) As Cr is added, the tensile strength gradually decreases with increasing quantity of Cr. However, the fracture toughness increases up to the point where the Cr addition is 0.32%. This is probably because precipitation of an equilibrium phase on the Cr group dispersed particles dominates and it distributes evenly inside the grain and relaxes a locally concentrated stress. However, if the addition is more than 0.32%, the number of the particle is too many and the volume ratio increases, which lowers the fracture toughness.

(3) In comparing addition of Zr and Cr, the tensile strength is superior in Zr added alloys. However, the fracture toughness is better in Cr added alloys. This is probably due to difference in the fracture modes.

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REFERENCES

[1] J. S. Santner, Met. Trans., 9A (1978), 768.

[2] Ohira & Kishi: Light Metals 34, (1984), 411.

[3] Kato, Ueda & Kobayashi: Light Metals 35, (1985), 359.

[4] Yoshida & Baba: Light Metals 31, (1981), 195.

[5] G. M. Ludtha & D. E. Laughlin: Met. Trans., 13A (1982), 411.

[6] Yoshida, Uno & Baba: Light Metals 34, (1984), 689.

[7] J. R. Rice, D. M. Tracey: J. Mech. Phys. Sol., 17 (1969), 201.

[8] D. S. Thompson: Met. Trans., 6A (1975), 671.

[9] Kobayashi, Kato, Shimizu & Ueda: Light Metals, 31, (1981), 248.

[10] Uno, Baba: Light Metals, 24 (1974), 216.

[11] J. R. Rice: Trans. ASME J. Appl. Mech. 35 (1968),378.

[12] M. F. Asyly: Phil. Mag., 14 (1966), 1157.

[13] T. Kawabata & O. Izumi: J. Mater. Sci., 11 (1976), 892

[14] T. Kawabata & O. Izumi: Acta Met., 24 (1976), 817).

[15] G. T. Hahn & A. R. Rosenfield: Met. Trans., 6A (1975), 653

[16] E. Hornbogen & K. H. Zungahr: Metallography 8 (1975), 181.